PB 189 343

AN ENVIRONMENTAL SURVEILLANCE LABORATORY FOR RADIONUCLIDE ANALYSES. PART I. ESTABLISHMENT OF AN ENVIRONMENTAL SURVEILLANCE LABORATORY: A CASE STUDY. PART II. ANALYSIS OF LARGE NUMBERS OF MILK SAMPLES FOR FISSION PRODUCTS

Edmond J. Paratta, et al

Public Health Service
Washington, D. C.

July 1964

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ENVIRONMENTAL HEALTH SERIES

Radiological Health

AN ENVIRONMENTAL SURVEILLANCE LABORATORY for RADIONUCLIDE ANALYSES

U. S. DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE

Public Health Service

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ENVIRONMENTAL HEALTH SERIES

This series of reports embraces the science and technology of creating and maintaining a healthful environment for man. Individual reports in the series deal with health factors in: air; water; food; chemical, biological, and radiological contamination; sound, vibration, and x-radiation; liquid, solid, and gaseous wastes; homes, places of employment and recreation, and institutions.

The principal subject area covered by each report in the series is identified by two letters appearing after the Public Health Service Publication No. "999," designating the series. The following letters and subject areas apply:

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WP - Water Supply and Pollution Control

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AN ENVIRONMENTAL SURVEILLANCE LABORATORY for RADIONUCLIDE ANALYSES

Part I - Establishment of an Environmental Surveillance Laboratory: A Case Study

Edmond J. Baratta
Donald C. Parks
Dade W. Moeller

Part II - Analysis of Large Numbers of Milk Samples for Fission Products

Marvin Rosenstein

U. S. DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE
Public Health Service
DIVISION OF RADIOLOGICAL HEALTH
Washington, D.C. 20201

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FOREWORD

In January 1964, Dr. Luther L. Terry, Surgeon General of the Public Health Service, released the text of several recommendations presented by the National Advisory Committee on Radiation. One of the recommendations, "that an active surveillance program be maintained by health agencies at the Federal, State, and local levels," lends special relevance to the two reports appearing in this Environmental Health Series publication.

The first of these two reports provides a record of experiences in developing and placing in operation the Northeastern Radiological Health Laboratory, one of the three regional laboratories established by the Division of Radiological Health for the surveillance of radioactivity in the environment. This report affords an insight for other agencies and groups into the resources that may be called upon and the details involved in designing and organizing an environmental surveillance facility. The study indicates how the various types of samples can be handled within the overall laboratory operation and outlines the procedures which have proven effective at this installation for managing the flow of samples on a large scale basis.

The second report provides an outline of some of the major operational considerations required for the analyses of fluid milk samples. The necessary requirements for space, equipment, and personnel are all related to the workload so that the values listed can be scaled upward or downward according to the size of the particular operation.

The two reports are offered as examples of how laboratory capabilities for radiological surveillance may be developed. It is recognized that special problems, including different scales of operations, will always arise and require individual solutions. However, it is hoped that the experiences presented here will be of help to individuals and groups faced with responsibilities for planning and establishing similar laboratories elsewhere.

ABSTRACT

The planning, establishment, and operation of a Public Health Service laboratory for radiation measurement and surveillance are described. Part I discusses the facilities, personnel requirements, equipment, and analytical capacities of the laboratory. Part II deals with the techniques of handling large numbers of milk samples for analysis of fission-product activity. Operational factors are considered, including intralaboratory quality control and time studies based on workload and laboratory capacity.

PART I

ESTABLISHMENT OF AN ENVIRONMENTAL SURVEILLANCE LABORATORY: A CASE STUDY

Edmond J. Baratta, Donald C. Parks, Dade W. Moeller

Introduction

Under a Presidential directive of 1959 (1), the Public Health Service was assigned additional responsibility with respect to the nationwide measurement and evaluation of environmental radiation levels. In meeting these responsibilities, the Division of Radiological Health made plans for the establishment of a number of regional laboratories to handle the required measurement and surveillance work. The first two of these were established in Las Vegas, Nevada, and Montgomery, Alabama, by the spring of 1960. The third laboratory was made possible by two events which occurred during August 1961:

1. Congress made "funds available for the establishment and operations of a laboratory in the Northeastern United States" (2).

2. The Atomic Energy Commission decided to terminate the operations of its laboratory in Winchester, Massachusetts.

The Northeastern Radiological Health Laboratory became a reality on October 12, 1961, when responsibility was transferred from the Atomic Energy Commission to the Public Health Service for the utilization of the Winchester facility. This building, approximately 10 years old, is situated on a 6-acre site and contains some 30,000 square feet of laboratory, plant engineering, and office space (3).

The purpose of this report is to review the salient factors involved in the establishment of this laboratory, particularly in those aspects related to the mass processing of environmental samples. This laboratory is a self-contained unit, larger than that required by most public and private organizations. Certain procedures, however, are basic regardless of the size of the installation. With this in mind, the various

components may be viewed as modules to be adopted or adapted by those responsible for establishing similar laboratories at the State or local level.

Program Planning

Although the analyses of large numbers of milk, water, total diet, and specific food samples were to form the backbone of the laboratory operation, early in the planning stages the decision was made to have basic programs in radiochemical research and radiological health engineering as associated activities. Because it was known that the laboratory building would be available several months prior to actual occupancy, a large part of the initial planning was done by the headquarters (Washington, D. C.) staff of the Division of Radiological Health, Public Health Service. This advance planning included the development of specifications for the long lead time electronic equipment, issuance of these for bid and award, preparation of detailed fiscal papers, establishment of staffing patterns, and selection of a senior staff. The officer-in-charge, senior chemist, and management officer were among the first personnel reporting to the Laboratory, and the detailed planning function was immediately transferred to the staff on-site. To assure continued close coordination with headquarters, however, joint program planning meetings formed a regular part of the operation throughout the first months of occupancy. Later this function became a part of the monthly senior staff meetings; consultation with headquarters continued, but on a less frequent and less formal basis.

Financing

As mentioned in the Introduction, the Division of Radiological Health had previously established similar laboratory facilities in Las Vegas, Nevada, and Montgomery, Alabama. The Northeastern Radiological Health Laboratory was patterned after these earlier installations. Reviewing this experience and taking into account the existing physical plant at Winchester, the Division made a single allotment available for capital equipment, supplies, personnel services, and other related costs

during the first 9 months of operation. This amount was to provide for the development of the basic capabilities required to fulfill the analytical service commitments of the Division within the 15-State area which the Laboratory was to serve (fig. 1).

With these guidelines, a working budget was developed which listed 31 positions to be filled during the first 9 months. Costs of other items (such as equipment, supplies, travel, and utilities) were also estimated. As would be expected, the largest single expenditure was for capital equipment, consisting primarily of radiation measuring systems.

Space Planning

In establishing this Laboratory one of the main problems was to fit the required program into the existing facility. The authors fully realize the tremendous advantage this situation provided. For example, in the establishment of two other laboratories by the Division of Radiological Health, many man-hours have been consumed in the planning, construction, and acquisition of suitable buildings.

As would be expected, the Laboratory at Winchester required a number of modifications to suit the specialized requirements of the Division of Radiological Health. Through early on-site visits by headquarters personnel and a review of similar operations at other laboratories, the necessary modifications were decided upon soon after acquisition of the building and immediate steps were taken to make the changes.

The AEC contractor previously occupying the building had been working with uranium ores, including the associated radium and other daughter products, so the building had to be surveyed completely to determine the extent of radioactive contamination. Several months of scrubbing, painting, and refinishing of laboratory furniture, equipment, pipes, and walls were required to make it acceptable for the intended low level measurements. This contamination, however, continued to plague the staff throughout the first year and it was not until all "hot" equipment was completely removed from the site that it ceased to be a problem. Until that time, in spite of precautions, contaminated equipment found its way back into the Laboratory from time to time or became a new

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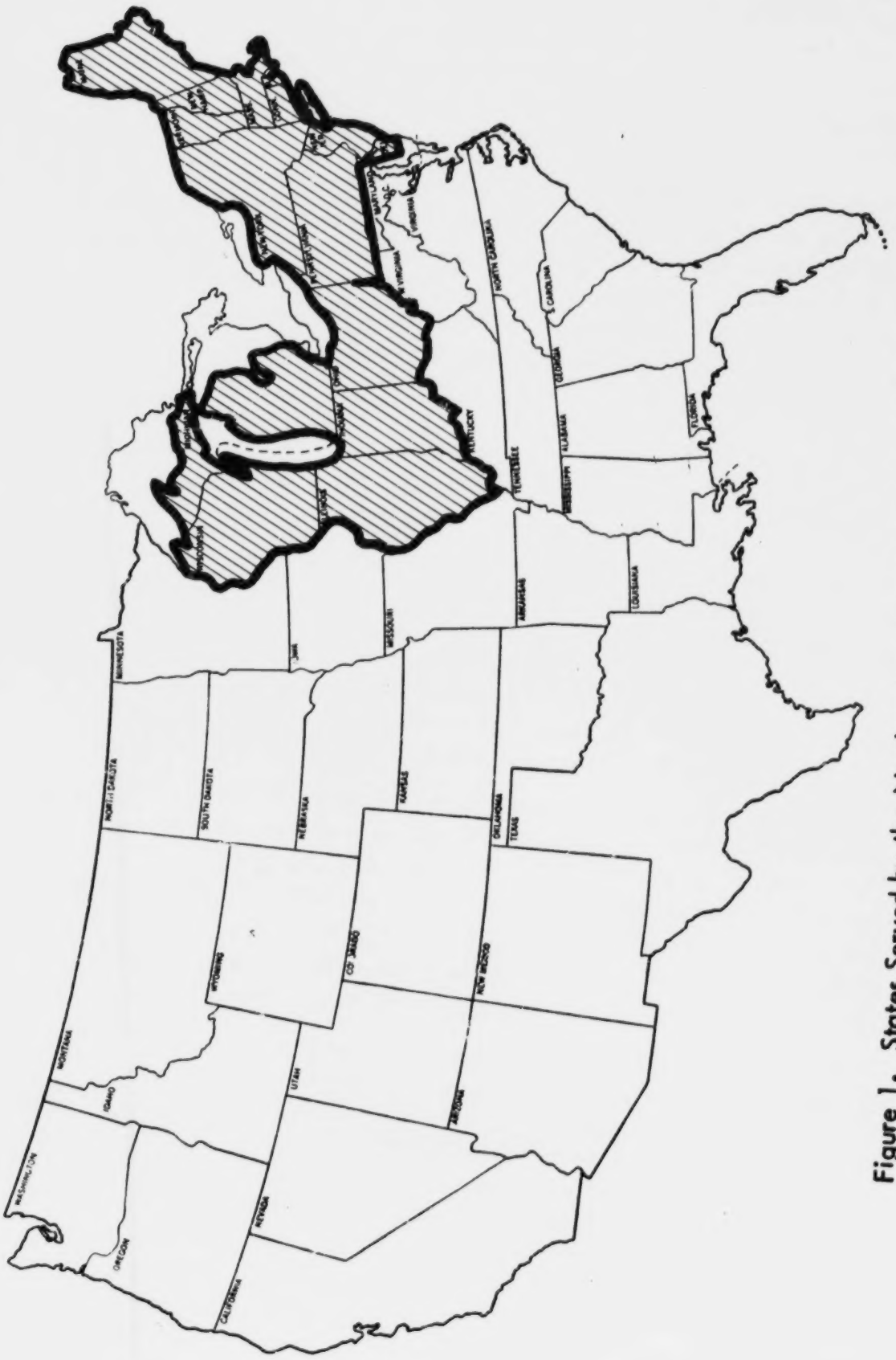


Figure 1. States Served by the Northeastern Radiological Health Laboratory

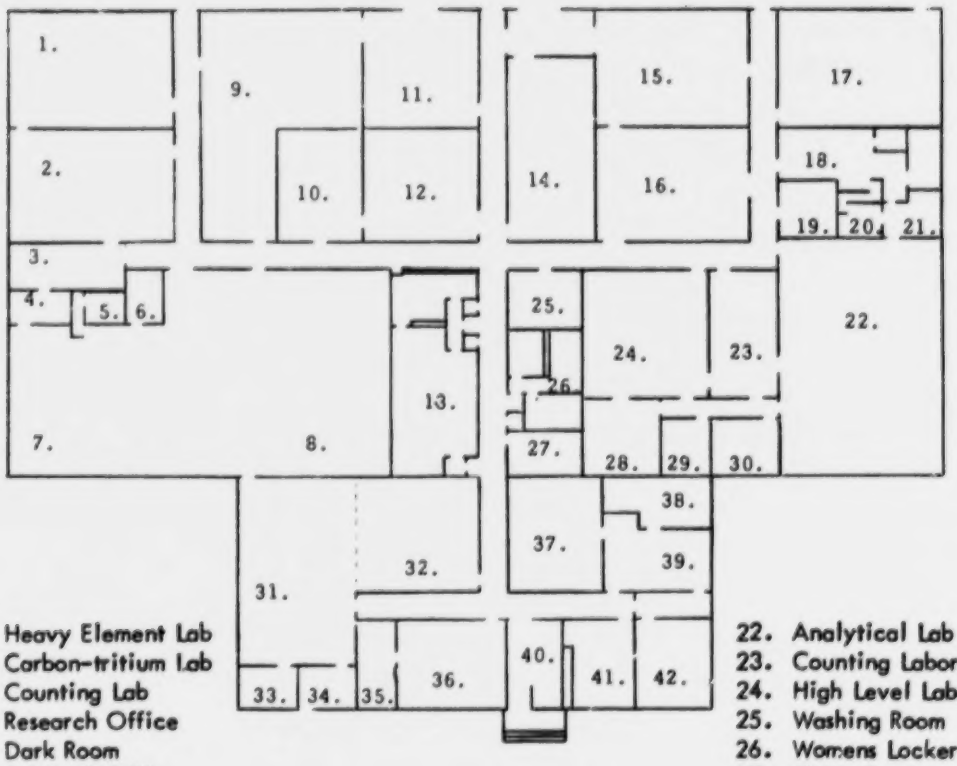
source when moved from one storage area to another.

Early in the planning, tentative allocations of space were made. Those shown on the floor plan (fig. 2) have proved quite satisfactory. The basic philosophy was to place each operation in the area where it was ultimately destined to be located. Although this created early situations in which only a few people worked in rather large areas, it avoided the necessity of constantly moving selected operations from room to room as new staff was added and occupied areas became crowded. This procedure also avoided delays in making the newer spaces operational even though, initially, it led to somewhat greater costs for utilities and maintenance.

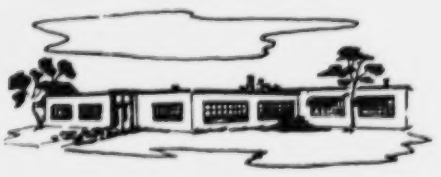
Maximum use was made of the existing facilities to minimize removing furniture or changing utilities. A major contribution to this plan was the opportunity to assign the working areas to each of the programs prior to the arrival of their respective staffs. There was no hesitation, however, in moving or removing installed equipment when it was located in one area but needed in another, or not needed at all.

Functions as Related to Layout

As may be seen from the floor plan (fig. 2), the Laboratory is self-contained. In the case of a laboratory established by a State, local, educational, or commercial agency, such facilities as a lecture room, dining room, library, conference room, reception office, building engineering services, and various management and personnel offices may be in existence to serve the laboratory in whole or in part. Thus the requirements for space, personnel, and services may be reduced considerably as compared to this case study. In addition, the range of analytical and research services that might be applicable to a newly established local laboratory would probably differ from those that characterize this laboratory. So long, however, as the planner keeps in mind the unit of operation he is undertaking, the approach outlined here should apply. Thus, for example, the establishment of a milk laboratory will require facilities for sample storage and preparation, spectroscopy, chemistry laboratories, and the availability of special analytical, engineering, and computer services. In addition, provision will have to be made for management, secretarial, and related functions.



1. Heavy Element Lab
2. Carbon-tritium Lab
3. Counting Lab
4. Research Office
5. Dark Room
6. Research Office
7. Electronics Lab
8. Engineering Lab
9. Pilot Plant
10. Sample Storage
11. Sample Reduction
12. Sample Preparation
13. Mens Locker Room
14. Stock Room
15. Boiler Room
16. Plant Engineering Shop
17. Milk Laboratory
18. Spectroscopy Lab
19. Plant Engineering
20. Dark Room
21. Analytical Office



SCALE
0' 15' 30'

22. Analytical Lab
23. Counting Laboratory
24. High Level Laboratory
25. Washing Room
26. Womens Locker Room
27. Phone Room
28. Methods Lab
29. Balance Room
30. Analytical Office
31. Lecture Room
32. Dining Room
33. Training Office
34. Personnel Office
35. Management Office
36. Management Operations
37. Library
38. Deputy OIC
39. Engineering Office
40. Reception
41. Conference Room
42. Officer in Charge

Figure 2. Floor Plan, Northeastern Radiological Health Laboratory

Similar considerations would also apply to the establishment of a food laboratory, heavy element laboratory, or other facility. In each case, advance program planning and consultation with appropriate Government agencies or other organizations are necessary. The experiences outlined in this report will, it is hoped, provide some insight into many factors involved in the planning of a surveillance facility.

Staffing

Concurrent with planning, financing, and physical plant preparation, was the problem of staffing. One of the first positions to be filled was that of the officer-in-charge. This individual was designated even before the building was occupied and, although he continued in another position for several months prior to reporting on site, he participated in the earlier planning stages. This procedure avoided the problems of the permanent person in charge having to take over from an acting chief who may have accomplished a large part of the initial work of setting up the Laboratory, with the possible attendant effects on staff morale.

The senior chemist played a major role in establishing the Laboratory through planning the purchase of required chemicals and laboratory processing and counting equipment, outlining necessary laboratory procedures, and interviewing and selecting professional and technical staff. He also handled their orientation and training as they reported for duty.

The management officer handled the many administrative details, such as installation of a telephone system, procurement of utilities, establishment of working relationships with other State and regional personnel offices, and initiation of the ordering, receiving, reporting, and accounting of supplies and equipment. He also had primary responsibility for recruitment and assisted in the overall formulation of Laboratory policies, particularly in reference to working rules and regulations.

Of importance in the early planning was the plant maintenance staff previously employed by the AEC contractor who operated the Laboratory. The familiarity of this staff with the building, its equipment, and its utilities proved invaluable in the decontamination operations and the removal and repair of existing equipment. This staff was also responsible for placing

a machine shop into operation after the building was acquired. The immediate availability of skilled craftsmen with lathes, drill presses, joiners, and welding apparatus was a real asset throughout the development phases of the Laboratory.

The hiring of most of the additional staff was conducted concurrently with the acquisition of clerical and secretarial personnel early in the recruitment operations. Particularly helpful in this case was the temporary assignment to the Laboratory of experienced secretarial help from headquarters. These people assisted in training new personnel as they reported, and in establishing routine office practices and procedures.

Another important and necessary position considered in the initial staffing was that of property clerk. This position requires the services of an individual familiar with property management and stocking, able to provide for immediate numbering and accounting of all unexpendable property and to maintain records of stocks ordered, received, and used. To prepare such records several months after operations have begun would be a time-consuming and tedious task.

The remaining positions in the staff were principally those of physical science aides, technicians, chemists, and administrative personnel. The acquisition and training of a good librarian, to provide reference materials to the professional staff, expedited the solution of many problems. Specific positions required in those phases of the Laboratory's operation pertaining to the mass processing of samples are shown on the following page. Experience has demonstrated that this staff was able to handle routinely 300 samples, requiring some 1,500 determinations, per month.

Orientation and Training

Since 90 percent of the current professional staff were introduced to the field of radiological health for the first time upon reporting to this Laboratory, short-term technical training has been an important part of the overall program since shortly after the facility became operational. Such training was accomplished initially by sending individuals to various training centers of the Public Health Service (4). Later, regular courses were offered on-site, thus permitting

Staffing Chart for Analytical Services Program

Function	Title
Laboratory Planning, Direction, and Control	* Laboratory Director Senior Chemist * Management Officer * Secretary
Sample Preparation	2 Chemistry Technicians
Milk Analysis	Chemist 2 Chemistry Technicians
Radiochemical Analysis	Supervisory Technician 3 Chemists 4 Chemistry Technicians
Radiation Measurement and Data Analysis	Mathematical Statistician Electronic Technician 2 Technicians (Counting Room) Clerk Typist
Administrative Services	Administrative Assistant Supply and Property Clerk Clerk Librarian Clerk Typist
Plant Engineering	Plant Engineer Mechanics Helper 3 Laborers
Total:	31 Staff Members

* It is estimated that approximately 50 percent of the time of these staff members is devoted to work related to sample analyses.

large numbers of the staff to participate. There was always the question of how many of the staff could be spared to attend, but because of the long-range dividends every effort was made to provide the opportunity to as many as possible. It should also be noted that training was not restricted to the professional staff. Several 2- and 3-day courses were also offered to the administrative staff; overall orientation programs were conducted for all employees new to the Federal employment system.

Sample Processing

The primary function of the Laboratory is to process environmental samples collected under the nationwide networks of the Public Health Service. These operations were placed in a category designated as the Analytical Services Program (fig. 3) and divided into the following four related group activities:

1. Sample preparation: The function of this group is to log in the samples and prepare them for gamma scanning and/or radiochemical analysis. For food and other solid items, gamma analysis requires that they be ground and homogenized. After gamma spectroscopy, the samples are returned to the preparation area for ashing prior to radiochemical separations.

2. Milk laboratory: This laboratory is responsible for the radiochemical analysis of all milk and milk products. Because of its importance as a source of human radionuclide intake, fluid milk represents one of the major items processed by this Laboratory.

3. General analytical laboratory: The staff in this laboratory is responsible for the analysis of all samples other than milk. During the first year of operation, these included bread, beef, lettuce, apples, coffee, margarine, eggs, and potatoes - all regularly collected under the Public Health Service surveillance system. In addition, this laboratory is responsible for any special samples, as well as a limited amount of analytical methods of development.

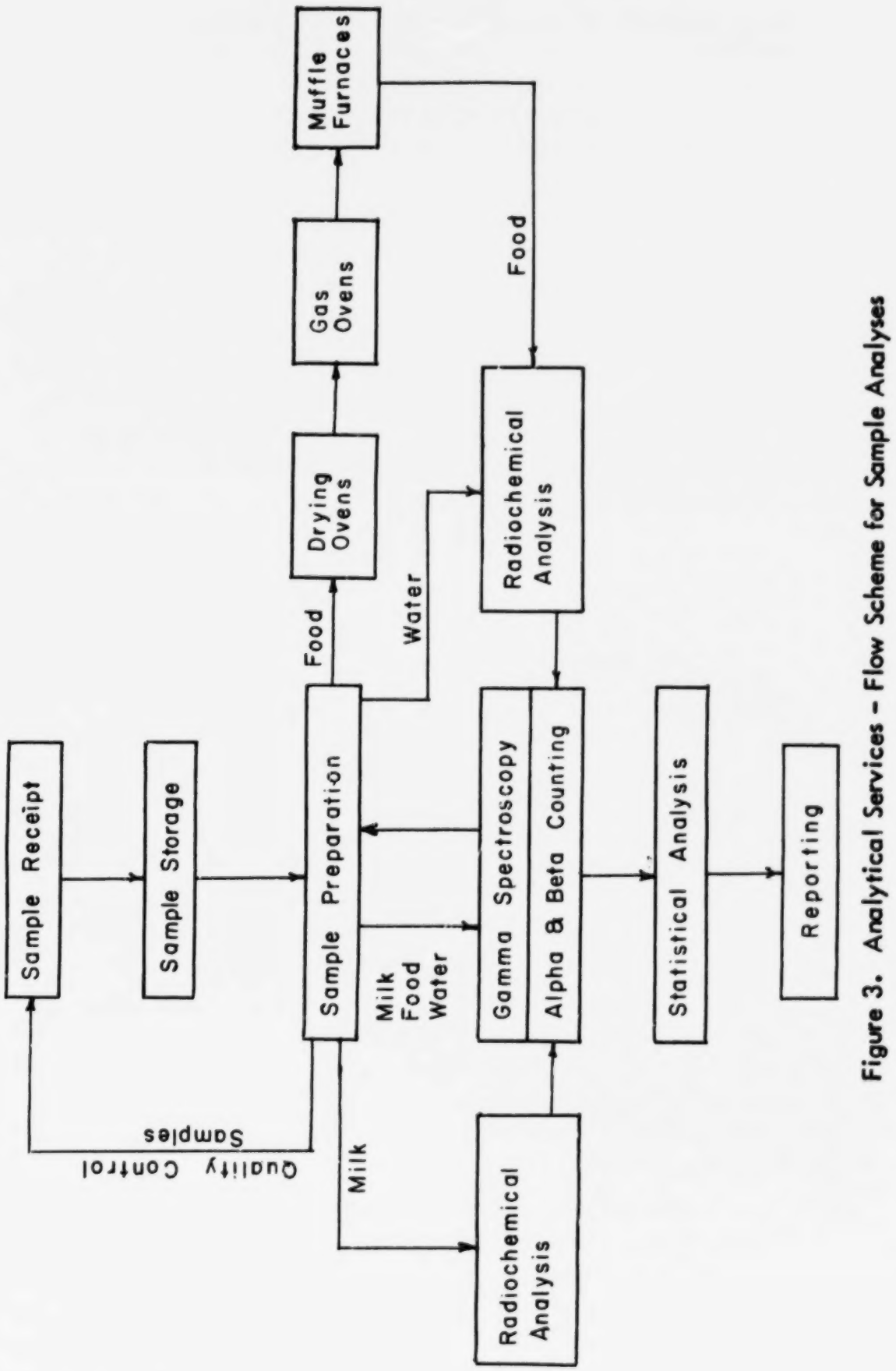


Figure 3. Analytical Services – Flow Scheme for Sample Analyses

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4. Counting room and electronics laboratory: After suitable physical and/or radiochemical preparation, all samples are submitted to the counting room staff for determination of their radioactivity content. This group is also responsible for the maintenance and calibration of the electronic equipment.

The Analytical Services Program also has two mathematicians on its staff to calculate and interpret data and to evaluate intralaboratory quality control.

Soon after the Laboratory began processing samples, a quality control program was established. Under this program, 20 percent of the samples are resubmitted for analysis as blind duplicates. As a measure of acceptable performance, limits of precision were established. Where lower performance is observed, immediate steps were taken to determine the source and to remedy the situation.

Equipment and Sample Analyses

Specifications, developed in connection with the other laboratories of the Division, were already available for securing electronic equipment for radiological analyses. Also available were basic lists of laboratory and administrative supplies, reference materials, and office furniture and equipment. Comparison of the workload at the other facilities with that proposed for this Laboratory permitted modification of these lists according to local needs.

Initially, facilities for the Analytical Services Program were planned on the basis of a monthly processing requirement of 1 ton of food and 150 samples of milk. These figures were especially important in estimating furnace capacity for ashing and in determining instrumentation requirements for sample counting.

1. Sample preparation: For ashing, a three-stage process was established. This consists of initial drying (stage 1 - 110°C), preliminary ashing (stage 2 - 300 to 400°C), and final ashing (stage 3 - 450°C and higher). With this approach, large samples (3 to 4 kilograms), containing very low levels of radionuclide contamination, could be completely ashed in 3 to 4 days.

Associated equipment for sample handling and processing includes a large cold-storage room, and equipment for grinding and homogenizing the samples.

After experimentation with equipment of several different types, a 5-horsepower meat grinder proved best for initial blending of the samples. This unit has the advantage of handling solid or heterogeneous samples of almost any type without requiring the addition of water.

The initial peeling of samples, such as potatoes and apples, is performed at a large stainless steel sink located in the middle of the room. Weighing balances, small specialized cutting equipment, and 1-gallon blenders for homogenizing samples round out the equipment for this phase of operations.

Initial drying (stage 1) of samples is handled in three wall ovens supplemented by two electric bench ovens having a total capacity of 90 cubic feet. For stage 2, two gas-fired ovens (39.4 cubic feet each) are used. Final ashing (stage 3) is accomplished in four electric muffle furnaces having a total capacity of 5.2 cubic feet.

2. Milk laboratory: Because milk can be processed in liquid form, its chemical analysis is handled in a separate laboratory. At this laboratory, an ion exchange procedure (5) is used to determine strontium-89 and strontium-90 in milk. Racks for passing samples in series through cation and anion exchange resin columns permit the handling of six samples daily on a production line basis. With a minimum of added equipment and manpower, the capacity could be increased to sixteen per day (eight columns on a two-shift basis). Because samples that are old, or have curdled, cannot be passed conveniently through the columns, the laboratory is also equipped to apply other analytical procedures where necessary (6).

3. Radiochemical processing: The chemical processing of all other samples is handled in the general analytical laboratory. Although primarily set up for total diet and specific food items, this laboratory also handles specialized samples, such as human hair and bones, drinking water, and biota. An auxiliary

laboratory provides space for a small staff responsible for solving problems encountered in routine analyses and for improving existing methodology. Because this group often employs radionuclide tracers in its work, it was located in a separate area from the main analytical laboratory (fig. 2).

4. Counting room: Equipment for the counting room was selected on the basis of the anticipated sample load and the specialized demands for counting. The basic units in the counting room (7, 8) are:

a. Two 400-channel pulse height analyzers (operated as four 200-channel units),

b. Four massive steel shields each housing a 4- by 4-inch NaI crystal,

c. Two low level beta counters with automatic sample changers,

d. Four shielded internal proportional counters for alpha and beta measurements,

e. Print-out systems for the beta and gamma equipment,

f. Desk calculators for routine data analyses.

Detailed data on the capacity of each of the units in this overall complex are listed in table 1. Counting times listed are average; specific values depend on such factors as the expected radionuclide concentration in the sample. In this regard, it is important to note that in time of heavy contamination (such as an emergency), when the sample load becomes high, the shorter counting period permitted would be compensating.

The installation and calibration of the counting equipment represent a major challenge to the staff of any new laboratory. Considerable assistance in this respect was made available to this Laboratory from other operating units in the Division of Radiological Health. By reducing the time required to train the local staff in the details of the operation of radiation measurement systems, such assistance enabled the Laboratory to produce meaningful data with a minimum of delay.

Table 1. Counting Time Capacity and Workload

Counting Time Capacity per Month*

GAMMA	hr/mo	BETA	hr/mo	ALPHA	hr/mo
2 "400" channel analyzers split into four 200-channel units	520	2 low-background units with automatic sample changers	880	4 single-chamber units	520

Counting Time Workload per Month

Normal workload per month (8/62)	GAMMA			BETA			ALPHA		
	No.counts	Min counted	Total hours	No.counts	Min counted	Total hours	No.counts	Min counted	Total hours
Milk	144	50	120**	288	30	144	20	100	33
Water	10	50	8	20	30	10	10	100	17
Food	70	50	58	100	30	50	50	100	83
Miscellaneous (thyroid, hair, honey, soil)	10	50	8	5	30	3	5	100	8
	234	@50	194**	413	@30	207	85	@100	141

* Based on 8.5 hour workday, 20 workdays/month.
2 hours/day allowed for instrument repair, background counting, etc.

** Not included are repeat analyses.

Based on past experience, the following features were sought for the counting room:

a. Limited access except to authorized personnel,

b. Pass boxes for receipt and release of samples,

c. Electric power from a source free of interference,

d. Controlled temperature and humidity,

e. Maintenance of positive pressure in the room.

Auxiliary Programs

Although the basic purpose of the Laboratory was the analyses of samples, certain auxiliary programs proved desirable in offering support for the primary objectives as well as providing a climate conducive to attracting and retaining competent employees. For these reasons, programs in research and engineering play an important role in the operation of this Laboratory. Although on paper each is a separate entity, they complement one another and provide the type of well-rounded program under which a professional staff thrives. The sample load is not always uniform, so specialized studies have also been developed within the staff of the Analytical Services Program. These, too, offer opportunities for leadership to the more outstanding employees and foster a stimulating environment.

In the course of development of the program, certain lessons were learned that may be usefully applied in establishing, maintaining, or reorganizing a laboratory. While it is not possible to foresee all contingencies and special requirements, it may be helpful to cite some examples of problems encountered and resolved.

In the procurement of materials and supplies, surplus property of governmental and other agencies can often be secured at substantial savings. Such sources were invaluable in the procurement of steel shielding, a fork lift truck, and other supplies. Savings can also be effected by obtaining certain items (such as cabinets, basins, food grinders, and

other items) from suppliers of kitchen furniture and hardware rather than from laboratory supply sources. The point is to find sturdy, serviceable equipment - whatever the source.

Experience has shown that certain facilities are extremely important. For example, it was quickly established that a constant voltage power source and efficient temperature and humidity controls were necessary for the counting room. In addition, adequate refrigerated storage space for samples proved to be a highly critical requirement.

Early in the operation of the NERHL facility, it was learned that the glassware ordered initially was insufficient for the volume of samples processed. Care should be exercised to avoid this kind of shortage, which can cause a bottleneck in the overall operations of the laboratory.

The institution of a quality control system is another important objective, to provide basic standards in the event of frequent personnel turnover. Experiences with the intralaboratory quality control program at the Laboratory justify its adoption.

With regard to library services, it is very often possible to supplement a laboratory's own facilities by establishing cooperative arrangements with university or industrial libraries. NERHL maintains such arrangements with the Massachusetts Institute of Technology and with some industrial firms.

Efficiency can be readily obtained in other areas also. For example, it has been found that while a chemist is needed to supervise radiochemical procedures, any such functions requiring the services of additional personnel can be performed adequately by technicians. Similarly, computer services need not be excessively complicated; even in a laboratory of the size of this particular case study, it has been found that one person using a hand-operated calculator can satisfactorily fill this need.

Comments and Conclusions

This report summarizes some of the experiences recorded by the authors who participated in the development of a regional laboratory of the Public Health Service. That the

staff received substantial assistance from within its own organization is readily acknowledged. It is hoped that this account will be of interest and help to others planning similar facilities at the State and local levels, or in educational institutions and industry. For almost any group, assistance in planning and designing a surveillance laboratory is available. The Division of Radiological Health through its regional office structure serves as a ready source of information and assistance to State and local agencies. Colleges and universities, in many cases, can turn for similar help to State organizations, and they, in turn, can request such aid from the Public Health Service. For industry, consultants are available to provide similar knowledge.

From the beginning of the project, a concentrated effort was made to keep local, State, and regional agencies informed of what was being done. Without exception, these organizations expressed interest in the work of the Laboratory and offered to assist in any way possible. A significant portion of this assistance was guidance on the establishing of sound, constructive relationships with the local community, schools, colleges, and news media. This resulted in excellent early radio, television, and newspaper publicity and expedited the securing of police and fire protection. The favorable impressions created by these means have continued to pay dividends during subsequent months.

A checklist of some of the items necessary in the establishment of a laboratory is given in the appendix. One key ingredient in the success of this particular project was the authority granted to the officer-in-charge to do everything possible, within the broad limits of Government and Agency regulations, to establish the facility in the shortest possible time and with a minimum of detailed direction.

As a consequence, it became possible for the Laboratory to achieve, within 1 year after the assignment of the first staff members to the project, a monthly processing rate of 300 samples involving 1,500 determinations.

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APPENDIX

Administrative Checklist on Starting of a Field Activity

1. Contact utility services and issue purchase orders for gas, electricity, water, and fuel oil, and for layout and installation of the telephone system.
2. Arrange for vehicles for transportation.
3. Personnel
    a. Establish positions for basic personnel (technicians, clerks, and professional and maintenance staffs).
    b. Arrange for securing rules and regulations for employing staff.
    c. Arrange for a staff member to become a notary public to execute legal documents.
    d. Secure basic forms for bringing personnel on duty.
4. Set up property accounting system.
5. Order maintenance and general supplies.
6. Secure dollies (small fork lift truck preferable) and tools for unloading, uncrating, and moving furniture, supplies, and equipment.
7. Obtain small safe for accountable documents, agent cashier funds, etc.
8. Designate staff member to be responsible for accountable documents.
9. Develop routine system for reporting progress and problems to headquarters and other appropriate offices.
10. Establish agent cashier fund.
11. Secure delegations of authority to handle travel, purchasing, special wearing apparel, drivers' licenses, and leaves.
12. Secure copies of basic operating manuals of the parent organization.

13. Establish blanket purchase arrangement for procurement of small items, such as:
    a. Hardware
    b. Chemicals
    c. Glassware
    d. Maintenance supplies
14. Secure audio-visual equipment.
15. Order long lead time items, particularly furniture, typewriters, and radiation measurement systems.
16. Obtain from a similar operating group a list of usual office supplies and forms, and issue orders for 6-month supply from appropriate sources.
17. Arrange for laboratory smocks and laundering service.
18. Rent post office box, or arrange for mail delivery.
19. Have sign for building painted and mounted; request directional sign posts at several nearby streets.
20. Prepare small informational brochure, including location (map), purpose, and program, address and telephone number of the laboratory.
21. Secure basic first-aid supplies and fire extinguishers.
22. Contact local police and fire departments and arrange for fire-alarm box directly to fire department and nightly and weekend checks of building and facilities.
23. Plan for physical maintenance and internal security of the building, and establish safety program.

PART II

ANALYSIS OF LARGE NUMBERS OF MILK SAMPLES FOR FISSION PRODUCTS

Marvin Rosenstein

Introduction

The significance of milk as an environmental medium for estimating radionuclide intake of the population is well established (1, 2). In order to obtain significant information on total population exposure from the radionuclide content of milk, a very large number of samples must be analyzed on a continuing basis. Naturally, the laboratory methods developed to determine the concentrations of specific nuclides in milk must be adapted to meet this need. The analysis of milk samples has become a large scale operation, and steps necessary for mass processing of these samples have had to be introduced.

Several analytical techniques are adaptable to mass processing of milk samples (3, 6). The techniques presently used at the Northeastern Radiological Health Laboratory (NERHL) are gamma scintillation spectroscopy and ion exchange. This report discusses the application by this Laboratory of these techniques to the milk analysis program of the Public Health Service Pasteurized Milk Network (2), as an example of mass milk analysis operations.

Technical Aspects of Milk Analysis

NERHL serves three regions of the U. S. Department of Health, Education, and Welfare in the national milk surveillance network: Regions I, II, and V. These regions comprise fifteen Northeastern States: Maine, Vermont, New Hampshire, Massachusetts, Connecticut, Rhode Island, New York, Pennsylvania, New Jersey, Delaware, Michigan, Ohio, Indiana,

Illinois, and Wisconsin. Twice weekly [1], State and local public health people in 20 selected large-population areas in these States collect milk samples. The collectors airmail a 1-gallon sample of pasteurized, homogenized milk, with formaldehyde added as a preservative, to the Laboratory. Each sample is a composite of milk from the larger dairies in a given area and contains that percentage of milk from each dairy which corresponds to the percentage of local population served by that dairy. In this manner about 90 percent of the milk consumed by the area under consideration is represented. The analytical results for these samples therefore provide useful estimates of the average concentration of radiocontaminants in the milk from any particular area.

Considerable experience (1) has established that relatively few of the great number of radionuclides occurring naturally or formed as a result of fission become incorporated in milk. Most of the possible radiocontaminants are eliminated because of the selective metabolism of the cow, which restricts gastrointestinal uptake and secretion into the milk. Only eight radionuclides are commonly found in milk in readily measurable amounts: of these only six are primary; the other two are formed in the milk as radioactive daughter products of two of the primary elements. Of the six primary radionuclides, one - potassium-40 - occurs in nature and is a normal and inevitable constitutent of all milk. It also accounts for the greater part of the total radioactivity present and thus, in a sense, acts as an "internal background" complicating the measurement of the manmade radionuclides.

The five manmade radionuclides that are primary milk contaminants are strontium-89, strontium-90, cesium-137, barium-140, and iodine-131. Strontium-90 and barium-140 have radioactive daughter products - yttrium-90 and lanthanum-140 - which are also therefore found in milk.

Three of these radionuclides - strontium-89, strontium-90, and yttrium-90 - are pure beta emitters, which must be separated from the bulk of the milk radiochemically for counting (1, 3, 4, 5). The other nuclides in a milk sample can be determined simultaneously by gamma spectroscopy (6).

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[1] Sampling schedules vary with fallout conditions; this frequency was in effect at the time the Laboratory began operations.

The procedure for the milk analysis is based on the occurrence of these, and only these, nuclides in milk samples. The flow chart for samples, as currently processed from time of collection to final tabulation of results, is presented in figure 1.

After receipt of the milk, a 3.5-liter aliquot of each sample is examined by gamma spectroscopy for several nuclides: Iodine-131, barium-140, cesium-137, and potassium-40. Counting time for this operation is 50 minutes, using a 4-by 4-inch sodium iodine (thallium-activated) crystal and multichannel analyzer. The data obtained from this procedure, together with background data similarly determined, supply the information from which the concentration of each gamma-emitting radionuclide in the sample can be calculated (6). Problems encountered in this phase of milk analysis are limited to equipment malfunctions and faulty calibration procedures, as no additional preparation or handling of the milk sample is necessary for gamma spectroscopy.

After gamma spectroscopy, the milk sample is stored under refrigeration for two weeks, during which time yttrium-90 is formed from the radioactive disintegration of strontium-90:

$$Sr^{90} \xrightarrow[28y]{\beta^-} Y^{90} \xrightarrow[64h]{\beta^-} Zr^{90}\ \text{(Stable)}$$

Yttrium-90 is formed from the radioactive disintegration of strontium-90 at such a rate that after 2 weeks the activity in the milk from yttrium-90 is essentially equal to the activity in the milk from strontium-90. This condition is called secular equilibrium (7). At the same time strontium-89 undergoes the following decay scheme:

$$Sr^{89} \xrightarrow[50.5d]{\beta^-} Y^{89}\ \text{(Stable)}$$

The formation of yttrium-90 from strontium-90 is an essential step in the radiochemical analysis of milk, because it provides a means of distinguishing the activity attributable to strontium-90 from the total activity of the combined strontium isotopes ($Sr^{89} + Sr^{90}$).

For milk, the radiochemistry involved in all the present analytical methods is basically the same. Where the several methods differ from one another is in the separation of the radionuclides of interest from the bulk of the milk. At this

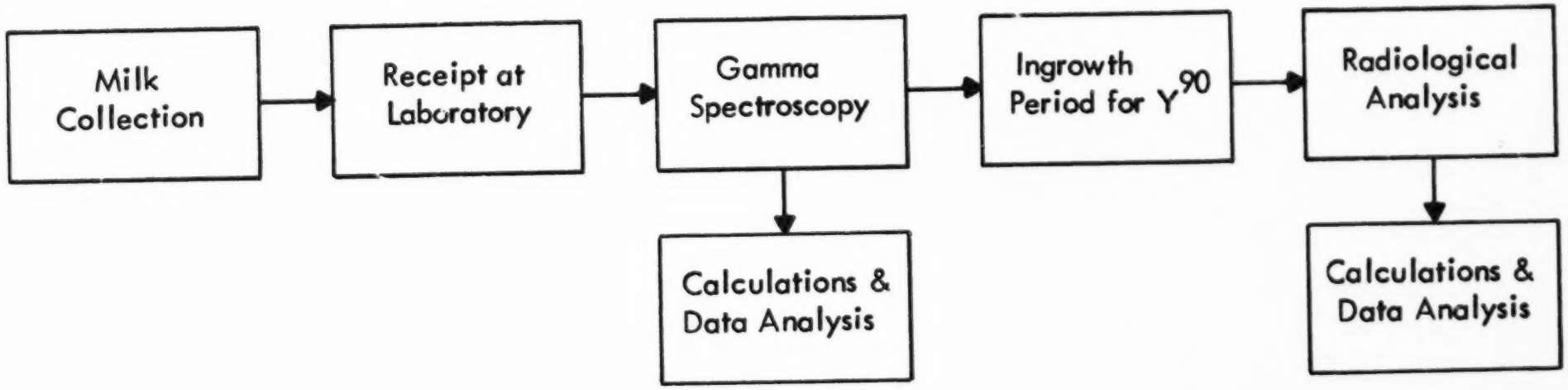

Figure 1. Analysis for Radionuclide Contaminants in Milk - Flow Chart

Laboratory, ion exchange resins are used for this purpose (3). This method is summarized in figure 2.

After the ingrowth period of 2 weeks, the two samples received from the same area in the same week are composited and 2 liters are submitted for radiochemical analysis. One liter of the milk is passed through cation and anion exchange columns in series; the other liter is kept as a duplicate sample in case the first sample should be accidently lost during analysis. Strontium ($Sr^{89} + Sr^{90}$) is isolated and counted for beta activity. An aliquot can be taken from the same solution, and stable calcium determined.

Yttrium-90 is adsorbed onto the anion resin from which it is later removed by displacement with hydrochloric acid solution. After subsequent chemical purification, it is also counted for beta activity. Since the yttrium-90 activity is equal to the strontium-90 activity under the condition of secular equilibrium, the concentration of strontium-90 in the milk can be calculated. By subtracting this value from the strontium ($Sr^{89} + Sr^{90}$) previously obtained, the amount of strontium-89 activity can be determined.

Two sets of reliable data are necessary to obtain the best results from the radiochemical separations. First, it is essential that the chemical compounds be recovered in pure form and in good yield, since the recovery data form the basis for final calculations, and errors in these data are compounded throughout the associated calculations. Second, proper counting procedures must be established and maintained. This involves calibration of equipment, routine counting techniques, and routine counting-equipment maintenance. Errors in counting are compounded in the final calculations. The two sets of data - chemical yields and counting statistics - are the greatest sources of error in the radiochemical determinations. Constant surveillance must be maintained over these factors to assure satisfactory final results.

Organizational Aspects of Mass Processing

In organizing a milk radionuclide analysis system, consideration must be given to two important factors. The results obtained from the milk analysis must be reliable, indicating that careful and proper analyses have been made. On the other

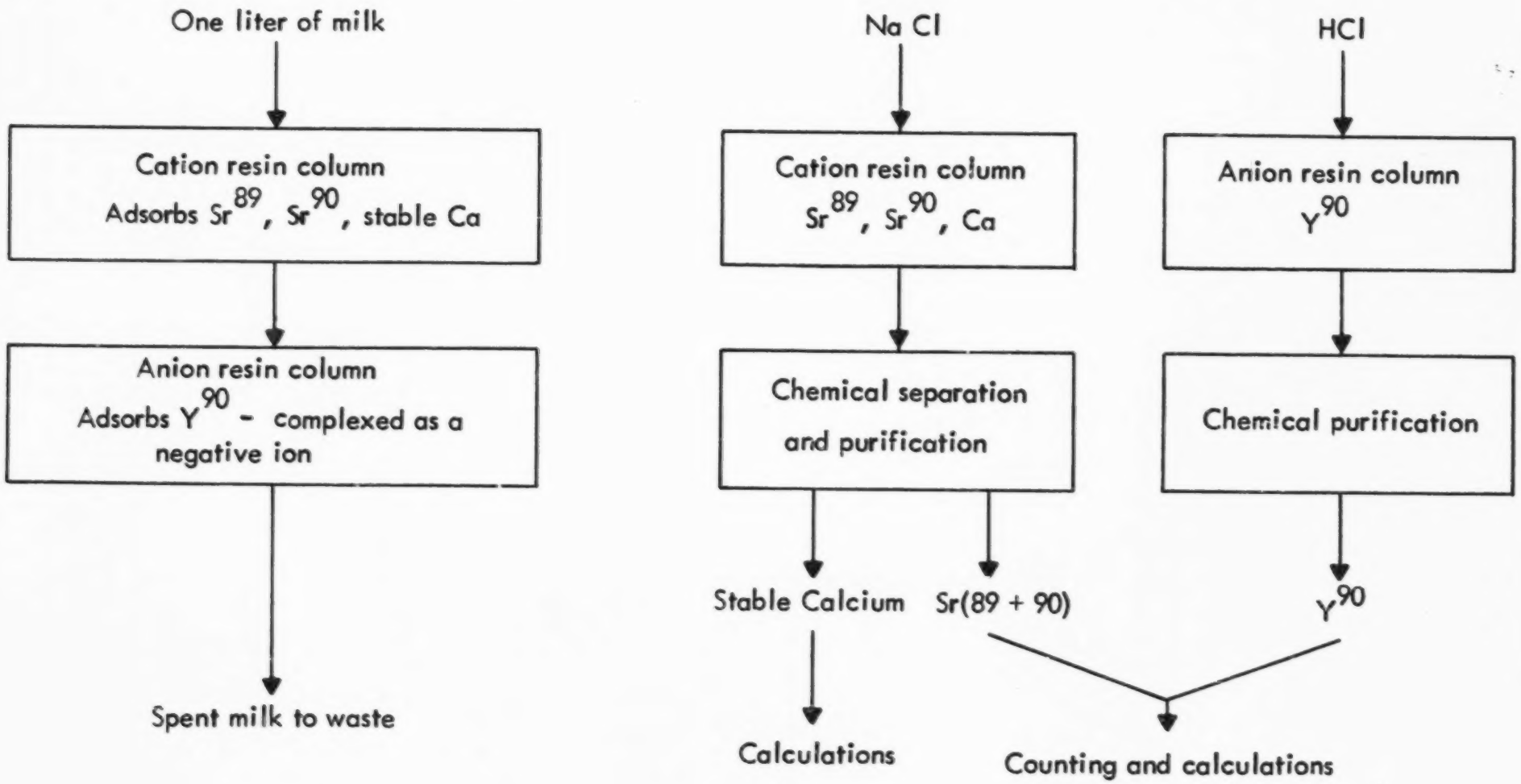

Figure 2. Summary of Ion Exchange Method

hand, it is equally necessary that large numbers of samples be processed, routinely and quickly, in order for the data to be adequate and reflect current conditions. Both of these factors must be weighed carefully in setting up procedures that will not compromise one or the other factor to the detriment of the final results.

Counting Time

A good example of balancing these two factors can be given in the determination of sufficient counting time in beta counting and gamma spectroscopy. The standard deviation in counting statistics for beta counting is given by the following relationship:

$$s = \left(R_o/t_o + R_b/t_b \right)^{1/2}$$

where s = standard deviation

R_o = observed counting rate (sample + background)

R_b = observed background counting rate

t_o = time over which observed counting rate is measured

t_b = time over which background counting rate is measured

Assuming that the background for the instrument is 1 count per minute, and the total counts per minute for the sample run is 14, counting errors can be calculated for various counting times. Data for 10-, 30-, 50-, and 100-minute determinations are summarized in table 1.

After making this type of inspection for both beta counting and gamma spectroscopy, sample load and desired sensitivity can be weighed so that the most efficient operation is realized. In the example cited, the counting error (9.4%) at the 10-minute counting time is too large to be acceptable. The time of 100 minutes is discarded because it is too long to permit the handling of a large number of samples. The two remaining times are both acceptable; but in the case of beta counting, a counting time of 30 minutes is the most reasonable solution, and in the case of gamma spectroscopy, the most acceptable counting time is 50 minutes.

Table 1. Counting Error as a Function of Counting Time

Counting time min	Standard deviation cpm	% Counting error *
10	± 1.22	± 9.4
30	± 0.71	± 5.5
50	± 0.55	± 4.2
100	± 0.39	± 3.0

* $\frac{\text{Standard deviation}}{\text{Net cpm (total - background)}} \times 100$

Intralaboratory Quality Control

To assure the integrity of results, an intralaboratory quality control program was introduced. Primarily, this type of control system was established to check the precision of laboratory results. It can bring to light any inconsistencies in the various procedures so that corrective measures may be taken. For example, after the gamma spectral data have been obtained, 20 percent of the samples, taken at random, are resubmitted to the counting room for control purposes. The technician does not know which samples are rechecks and which are not. The results are then compared with those originally reported. In a like manner, 25 percent of the milk network samples are resubmitted for radiochemical analysis. Here again, the original sample which the control represents is unknown to the technician. The results of this type of intralaboratory quality control program have proved very useful in evaluating procedures and data.

Chemical Processing

To facilitate handling the large number of milk samples received at the Laboratory, two organizational steps were taken. First, the time needed to perform many of the steps in the radiochemical analysis was reduced by mechanical means: For example:

1. Eight samples are run simultaneously, one in each of eight separate ion exchange systems.

2. A trough is installed to permit the discarding of effluent liquids which are not needed for further analysis and require no further attention.

3. A system of hot plates is provided which allows the technicians to collect the effluents from the cation colums and heat them at the same time. The apparatus is shown in figure 3.

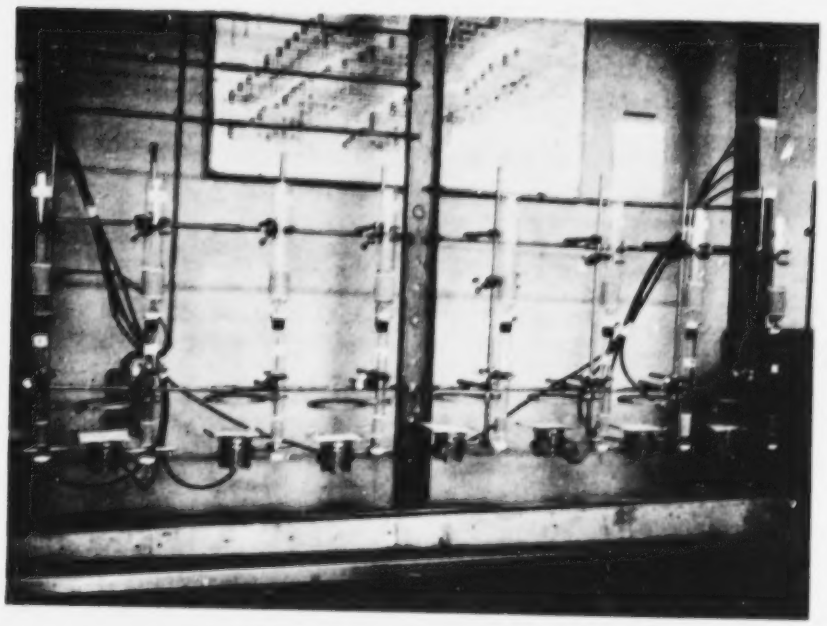

Figure 3. Ion Exchange Apparatus

Second, the responsibilities of analysis can be divided among personnel to facilitate handling as summarized below.

1. Gamma spectroscopy is performed by technicians under the direct supervision of the director of Analytical Services. One of the duties of the Mathematics and Statistics group is to conduct the necessary calculations for gamma spectroscopy.

2. Radiochemical analysis is carried out as follows:

   a. The first stages of the ion exchange method are attended to by two technicians and a chemist in charge of the

milk radiochemistry laboratory.

b. After separation of the columns, each technician performs one analysis, whether it be the total strontium ($Sr^{89} + Sr^{90}$) analysis or the strontium-90 analysis, on all samples for the day.

c. Calcium analysis of samples is done by whichever technician is available.

d. For final beta counting the samples are submitted to the counting room at the end of the day, for counting during the night, using an automatic sample changer.

e. All calculations on radiochemical analyses performed by the milk chemistry laboratory staff are handled by the chemist in charge so that he may review the data for gross errors in procedure.

Such a division of responsibilities has proved to be both timesaving and practical.

Operational Aspects of Mass Processing

The number of samples received from the collection stations represents a large quantity of analytical work. By applying the procedures discussed above, considerable increases in the number of samples that can be processed have been achieved. The milk workload for 1 week, under normal conditions, can be divided into two categories: gamma spectroscopy and radiochemical analysis. As shown in table 2, samples received from the milk network stations basically establish the workload of the milk analysis laboratory, but the workload is augmented by sufficient control samples to study the reproducibility of results. The figures in table 2 are based on conditions existing at the time of this report and will vary depending on environmental radionuclide contamination patterns.

Table 2. Milk Sample Load for One Week - NERHL

Category	Number of milk network stations	Network samples	Control samples	Total weekly load
Gamma spectroscopy	20	40	8	48
Radiochemical analysis	20	20*	5	25

*This figure represents composite of the two weekly samples from each station.

To allow for possible increases in workload, the Laboratory was designed to handle a considerably larger number of samples than are presently received. Giving proper consideration to problems encountered during normal analytical procedures, the capacities of the Laboratory in the same categories as described above are as shown in table 3. As would be expected, the ability to process samples is dependent on the amount of operable equipment. Therefore, the capacities shown in table 3 could be readily expanded by further addition of analytical equipment.

Table 3. Analytical Capacity for Milk Samples

Category	Analytical unit	Capacity per unit		No. of units	Total capacity	
		per day	per week		per day	per week
Gamma spectroscopy	Shield and crystal	5	25	4	20	100
Radio-chemical analysis	Ion exchange system	1	5	8	8	40

Of importance also, especially in mass processing, is the time element needed to successfully perform the analytical operations. A time study was conducted in connection with all phases of the milk analysis. Figure 4 shows the results of this study for eight determinations.

It can be seen from the study that the total elapsed time of analysis from receipt of the sample to final calculation is approximately 2 weeks. Note should be made, however, of the fact that gamma analysis is complete after the first day, and beta analysis is complete within one and one-half days after the ingrowth period. If necessary, total elapsed time can be reduced considerably by shortening the ingrowth period. Approximate results can be obtained after a 5-day ingrowth period if desired.

The following general conclusions can be drawn regarding personnel and equipment.

1. One technician operating one gamma spectrometer can easily scan 20 milk samples a week, and could handle up to 25 milk samples a week.

2. The same technician can perform all the routine calculations and procedures involved in gamma spectroscopy.

3. The same technician can operate the low level beta counting equipment for all samples from radiochemical analysis.

4. One chemist, and an assistant technician, using four ion exchange systems and related laboratory glassware, can perform all the chemical analyses and calculations for 20 milk samples a week.

5. To increase the workload from 20 to 40 samples a week, four additional ion exchange systems and a second technician would be needed.

Therefore, if a low level analytical facility has been established, and basic counting instrumentation is available, the additional equipment and personnel needed to carry out a functional milk analysis program is modest.

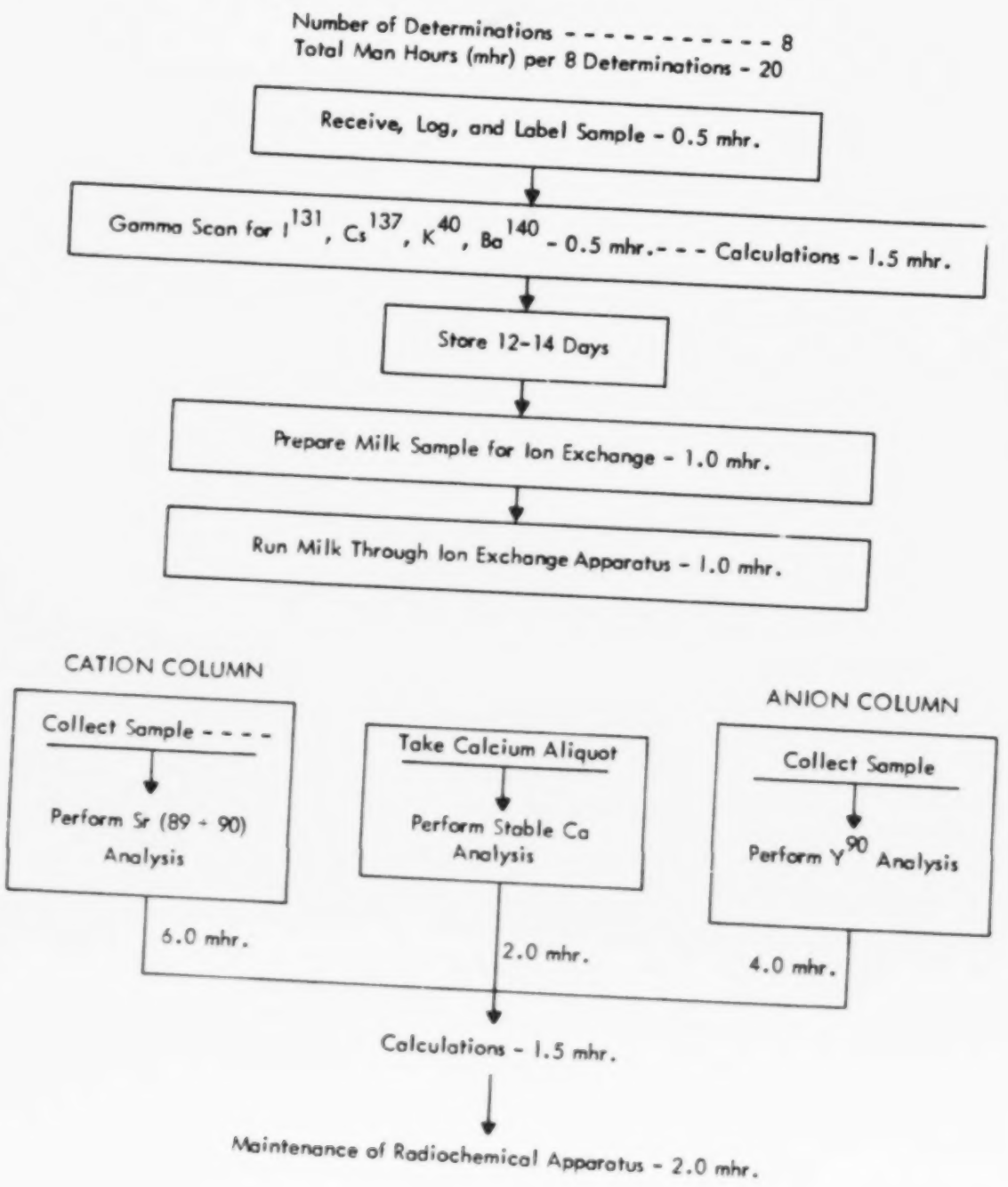
Number of Determinations - - - - - - - - - - - 8
Total Man Hours (mhr) per 8 Determinations - 20
Receive, Log, and Label Sample - 0.5 mhr.
Gamma Scan for I131, Cs137, K40, Ba140 - 0.5 mhr. - - - Calculations - 1.5 mhr.
Store 12-14 Days
Prepare Milk Sample for Ion Exchange - 1.0 mhr.
Run Milk Through Ion Exchange Apparatus - 1.0 mhr.
CATION COLUMN
Collect Sample - - - -
Perform Sr (89 + 90) Analysis
6.0 mhr.
Take Calcium Aliquot
Perform Stable Ca Analysis
2.0 mhr.
ANION COLUMN
Collect Sample
Perform Y90 Analysis
4.0 mhr.
Calculations - 1.5 mhr.
Maintenance of Radiochemical Apparatus - 2.0 mhr.

Figure 4. Time Study of Milk Analysis

Summary

The need for extensive data in determining radionuclide intake in man has necessitated mass processing of milk samples. For a mass milk processing program to be an efficient operation, from which reliable data can be obtained, existing technical procedures must be adapted to large numbers of samples; and personnel as well as laboratory operations must be arranged in a manner to produce the most efficient operation. In particular, the most important factors in a mass processing milk program are:

1. Collection of a representative sample for analysis,
2. Application of gamma spectroscopy techniques to fluid whole milk samples,
3. Application of radiochemical analyses for beta-emitting nuclides in milk,
4. Establishment of a control system for data produced,
5. Knowledge of the sample workload required,
6. Knowledge of the laboratory capacity desired,
7. Study of the time element involved in operations,

The methods used by NERHL are presented as an example of the application of these elements to a mass milk analysis program. It is hoped that the various State and local radiological health laboratories will find the information in this paper useful in planning programs of mass milk analysis for fission products.

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